

9. P807308

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
31 May 2001 (31.05.2001)

PCT

(10) International Publication Number  
**WO 01/38861 A2**

(51) International Patent Classification<sup>7</sup>: **G01N 23/00**

(21) International Application Number: **PCT/GB00/04452**

(22) International Filing Date:  
23 November 2000 (23.11.2000)

(25) Filing Language: **English**

(26) Publication Language: **English**

(30) Priority Data:  
9927555.4 23 November 1999 (23.11.1999) **GB**

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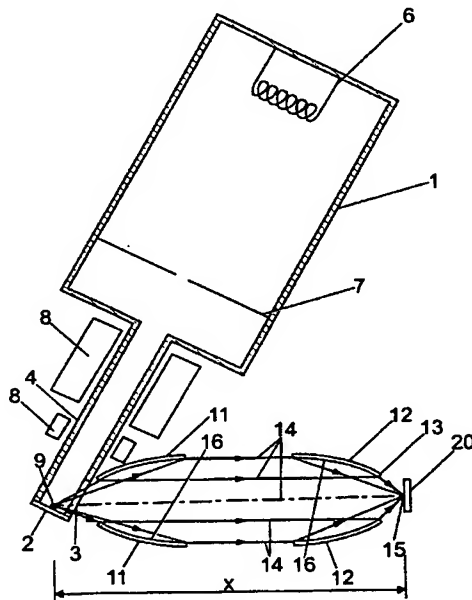
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(81) Designated States (*national*): AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European

[Continued on next page]

(54) Title: **X-RAY FLUORESCENCE APPARATUS**



(57) Abstract: This invention relates to a portable apparatus for carrying out X-ray fluorescence spectrometry on specimen materials at a distance from the apparatus. The apparatus comprises an X-ray generating tube (1), such as a microfocus tube, and two paraboloidal X-ray reflecting mirrors. The first collecting mirror (11) is positioned in close coupled arrangement adjacent to the exit window (3) of the tube (1), such that it emits parallel X-ray radiation (14) to the second focusing mirror (12) which is aligned on the axis of and spaced apart from the first mirror (11). The second mirror (12) collects the parallel X-ray radiation (14) at its end closest to the first mirror (11) and emits X-ray radiation in a focused beam onto the specimen (20). The distance between the first and second mirrors is adjusted to suit the distance from the X-ray tube (1) to the specimen (20). Focal spots on the specimen (20) of diameter less than 15  $\mu\text{m}$  are possible, enabling precise analysis of small areas of the specimen.

WO 01/38861 A2



patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

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- *Without international search report and to be republished upon receipt of that report.*

1     **X-ray Fluorescence Apparatus**

2

3     This invention relates to an apparatus and method for  
4     carrying out X-ray fluorescence spectrometry (XRF),  
5     and particularly to a portable apparatus which is  
6     able to generate X-ray fluorescence in materials at a  
7     distance from the apparatus.

8

9     X-ray fluorescence spectrometry is a non-destructive  
10    technique for determining the elemental composition  
11    of a wide variety of materials. X-ray fluorescence  
12    (XRF) is the secondary emission of X-rays at  
13    wavelengths characteristic of each element present  
14    when a material is irradiated with a primary X-ray  
15    beam. In commercially available XRF spectrometers  
16    the bulk sample is usually irradiated directly by X-  
17    rays from a sealed tube. The technique is  
18    sufficiently sensitive to detect elements which are  
19    present at concentrations as low as one or two parts  
20    per million. There is however a requirement for

1 greater sensitivity in applications in which it is  
2 desired to examine small areas on bulk samples or  
3 where the sample itself is small. The type of  
4 instrumentation required for this technique is  
5 sometimes called Micro X-ray Fluorescence Analysis  
6 (MXRFA or MXA) apparatus.

7  
8 Several methods presently exist for MXRFA. Among  
9 them is the use of mono-capillary and poly-capillary  
10 X-ray focusing optics coupled to standard or  
11 microfocus X-ray generating tubes. These suffer from  
12 the drawback that samples have to be placed very  
13 close to the output of the optic (generally less than  
14 300  $\mu\text{m}$ ). The minimum focal spot generally  
15 commercially available with polycapillaries is 28  $\mu\text{m}$ .  
16 This is relatively large and limits the fineness of  
17 the resolution with which areas of a sample can be  
18 analysed.

19  
20 Another method which presently exists for MXRFA is to  
21 use a synchrotron in conjunction with Fresnel lenses.  
22 Such apparatus is massive and not portable, although  
23 beams having a focal spot of only 1  $\mu\text{m}$  can be  
24 achieved, giving greater accuracy in analysis of  
25 samples. This method suffers from the disadvantage  
26 that synchrotron radiation sources are large fixed  
27 facilities which are not portable and are not  
28 available in most laboratories, so cannot be accessed  
29 on a routine basis.

30

1 A further method of MXRFA which exists is the use of  
2 a synchrotron in conjunction with mono-capillary  
3 lenses. Such apparatus is also not portable, and  
4 beam sizes are limited to a focal spot of 5-10  $\mu\text{m}$ .

5

6 It is therefore an object of the invention to provide  
7 an apparatus for carrying out X-ray fluorescence  
8 spectrometry which is portable yet which is capable  
9 of analysing samples of are less than 30  $\mu\text{m}$ .

10

11 According to a first aspect of the present invention  
12 there is provided an apparatus for carrying out X-ray  
13 fluorescence spectrometry comprising an X-ray  
14 generating tube and two paraboloidal X-ray reflecting  
15 mirrors, the generating tube having an X-ray source  
16 and an X-ray exit window through which X-ray  
17 radiation from said source is emitted,  
18 the first mirror being aligned on a first axis and  
19 positioned in close coupled arrangement adjacent to  
20 the exit window, the second mirror being aligned on  
21 said first axis and being positioned in spaced apart  
22 relationship to the first mirror,  
23 the first mirror being adapted to collect diverging  
24 X-ray radiation at its first end adjacent to the  
25 collecting window and to emit X-ray radiation in a  
26 substantially parallel beam at its second end,  
27 the second mirror being adapted to collect  
28 substantially parallel X-ray radiation at its first  
29 end closest to the first mirror and to emit X-ray  
30 radiation in a focused beam at its second end.

31

1 By using first and second mirrors in this way, the  
2 focal spot on the target of the X-ray tube is  
3 transferred to the image plane, at unity  
4 magnification. The focal spot at the image plane on  
5 the sample subjected to fluorescence has a high  
6 brightness, and focal spots on the sample of diameter  
7 less than 15  $\mu\text{m}$  are possible.

8  
9 Preferably the first and second mirrors are  
10 cylindrical specularly reflecting mirrors.  
11 Preferably the first end of the first mirror is  
12 positioned between 5 and 50 mm from the X-ray source.

13  
14 Preferably the apparatus further comprises a housing  
15 containing the first and second mirrors.

16  
17 The second mirror may be fixed in position relative  
18 to the first mirror.

19  
20 Alternatively the second mirror may be movable in  
21 position relative to the first mirror. The apparatus  
22 may further comprise a guide means for guiding said  
23 second mirror in a direction parallel to the first  
24 axis, and adjustment means for adjusting the spacing  
25 of the first and second mirrors.

26  
27 The apparatus may further comprise angular adjustment  
28 means adapted to allow angular adjustment of the  
29 mirror housing with the X-ray generator tube.

30

1 Preferably the X-ray generator tube is adapted to  
2 produce an X-ray source at the target having a  
3 maximum width of less than 50  $\mu\text{m}$ , more preferably  
4 less than 15  $\mu\text{m}$ .

5  
6 According to a second aspect of the present invention  
7 there is provided a method of delivering X-ray  
8 radiation to a specimen for the purpose of X-ray  
9 fluorescence spectrometry using an X-ray generating  
10 tube, the generating tube having an X-ray exit window  
11 through which X-ray radiation is emitted,  
12 the method comprising placing first and second  
13 paraboloidal X-ray reflecting mirrors between the  
14 exit window and the specimen,  
15 using the first mirror to collect diverging X-ray  
16 radiation at its first end adjacent to the exit  
17 window and to emit X-ray radiation in a substantially  
18 parallel beam at its second end,  
19 and using the second mirror to collect substantially  
20 parallel X-ray radiation at its first end closest to  
21 the first mirror and to emit X-ray radiation at its  
22 second end to a focused spot on the specimen.

23  
24 Preferably the method uses an apparatus according to  
25 the first aspect of the invention.

26  
27 Embodiments of the invention will now be described,  
28 by way of example only, with reference to the  
29 accompanying figures, where:

30

1 Fig. 1 is a schematic view of two X-ray focusing  
2 mirrors used in accordance with the invention to  
3 focus an X-ray beam from the source on the X-ray  
4 target to the sample to be subject to X-ray  
5 fluorescence spectrometry;

6

7 Fig. 2 is a schematic view of an apparatus according  
8 to a first aspect of the invention having mirrors  
9 fixed relative to each other; and

10

11 Fig. 3 is a schematic view of an apparatus according  
12 to a second aspect of the invention having mirrors  
13 adjustable relative to each other.

14

15 Referring to Fig. 1 there is shown, in a schematic  
16 form and not to scale, an X-ray generating tube 1  
17 having an exit window 3, an electron source 6, an  
18 anode 7, focusing and stigmator coils 8 and a target  
19 2 on which is formed an X-ray source 9. A suitable  
20 X-ray generating tube is the MICROSOURCE™ tube  
21 described in International Patent Application No  
22 PCT/GB97/02022, which is a compact X-ray generator  
23 capable of producing small-size, high intensity X-ray  
24 sources for low power input. Typically the exit  
25 window 3 of the generator 1 is provided in the narrow  
26 portion 4 of the X-ray tube about which the X-ray  
27 focusing coils 8 are arranged, to the side of the X-  
28 ray target 2. A first X-ray focusing mirror 11, the  
29 collection mirror, is positioned adjacent to the exit  
30 window 3 in close coupled arrangement, and a second  
31 X-ray focusing mirror 12, the focusing mirror, is



1 arranged coaxially with the first X-ray focusing  
2 mirror 11, to transfer the . Suitable mirrors 11, 12  
3 are MICROMIRROR™ X-ray optics as supplied by Bede  
4 Scientific Instruments Ltd. The mirrors are  
5 cylindrical specularly reflecting mirrors. Each  
6 mirror comprises a cylindrical body having an axially  
7 symmetrical passage extending therethrough. There is  
8 an aperture at each end of the body which  
9 communicates with the passage. The reflecting  
10 surface is on the inside of the long axis of the  
11 cylinder and has a shape corresponding to a  
12 paraboloid of revolution about the long axis of the  
13 cylinder.

14

15 A paraboloidal profile produces an almost parallel,  
16 essentially non-divergent beam 14. The interior  
17 reflecting surface 16 is coated in an exceptionally  
18 smooth coating of gold or similar in order to provide  
19 specular reflectivity. Typically the mirror is made  
20 of nickel and is of the order of 10 to 100 mm in  
21 length, typically about 30 mm. The outside diameter  
22 of the mirror is typically 6 mm. The internal  
23 diameter is typically less than 4 mm. The entry  
24 aperture is generally smaller than the exit aperture.

25

26 The two mirrors have an identical profile. The  
27 source to first mirror distance is in the range 5 to  
28 50 mm.

29

1 Typically the X-ray generator produces a sub-15  $\mu\text{m}$   
2 spot source on a target of less than 10 mm diameter  
3 at a power of up to 30 W.  
4

5 The first mirror or paraboloidal optic 11 has a high  
6 angle of collection and reflects X-rays into a  
7 substantially parallel beam. In practice a beam of  
8 divergence less than 40 arc seconds can be achieved.  
9

10 The second mirror or paraboloidal optic 12 takes the  
11 parallel beam and focuses it down to a spot 15 on the  
12 specimen 20 of a size similar to that of the X-ray  
13 source, typically a spot with a diameter of less than  
14 15  $\mu\text{m}$ .  
15

16 The focus 15 of the second optic 12 is typically  
17 about 10 to 20 mm away from the far end 13 of optic,  
18 giving a much more convenient working distance than  
19 is available from prior art XRF apparatus, such as  
20 monocapillaries.  
21

22 The distance between the two optics 11, 12 may be  
23 continuously changed without affecting the focal spot  
24 quality, thereby allowing a range of source to sample  
25 distances X to be achieved. Typically distance X  
26 will be 100 mm or more.  
27

28 X-ray optics have very well defined profiles and low  
29 surface roughness, and therefore work at very high  
30 efficiency. By using paraboloidal mirrors the  
31 apparatus of the invention achieves broad band

1 transmission of X-rays, with an efficiency close to  
2 1, since only double reflection of the X-ray  
3 radiation is required.

4

5 The invention achieves high X-ray brightness at the  
6 focal plane on the target, with a focal spot diameter  
7 of as low as 10  $\mu\text{m}$ .

8

9 The apparatus of the invention is truly portable,  
10 giving it applications in areas such as forgery  
11 detection, which require the apparatus to be taken to  
12 the specimen.

13

14 A parabolic surface will produce a parallel beam if  
15 the source is placed at the focal point. Conversely  
16 a focused beam will be brought to a focus when a  
17 parabolic surface is illuminated with a parallel  
18 beam. Therefore the method and apparatus of the  
19 invention serves to transfer the image of the X-ray  
20 spot from the target to the specimen. It should be  
21 noted that the target may not be perpendicular to the  
22 axis of the of the mirrors, so that the effective  
23 dimension of the image on the target, when viewed  
24 along the axis of the mirrors, is less than the  
25 actual dimension on the target.

26

27 The focal spot size at the specimen is thus primarily  
28 determined by the spot size on the target of the X-  
29 ray tube. Since the first mirror produces a parallel  
30 beam, the focal spot size at the specimen is, within  
31 practical limits, independent of the distance of the

1 second mirror along the beam axis. Therefore the  
2 second mirror can be placed at the required distance  
3 from the first in order to suit the geometrical  
4 requirements of the equipment.

5  
6 Figs. 2 and 3 show two schematic arrangements for  
7 housing the apparatus of the invention.

8  
9 In the simplest case, shown in Fig. 2, the collector  
10 and focusing mirrors 11, 12 are aligned with each  
11 other and are fixed within a cylindrical housing 30.  
12 The housing is aligned relative to the X-ray source  
13 9, shown purely schematically in Figs. 2 and 3, on  
14 the beam axis 32, either fixedly or adjustably. The  
15 housing 30 may be subject to a partial or total  
16 vacuum, to improve the efficiency of the mirrors and  
17 reduce energy absorption as the X-rays pass through  
18 the gas in the housing 30. It is to be understood  
19 that in practice the source 9 is part of an X-ray  
20 generating tube 1 (not shown in Figs. 2 and 3).

21  
22 In use the housing 30 is placed adjacent to the X-ray  
23 source, and a control mechanism 35 allows fine  
24 adjustment of the position of the housing 30 in the  
25 x, y and z directions so that the axis 32 of the  
26 mirrors is accurately aligned with the X-ray source 9  
27 and directed to the specimen 20. The control  
28 mechanism 35 may comprise any suitable mechanisms  
29 which permit fine translational adjustment, such as  
30 lead screws or Vernier controls.

31

1 In the example of Fig. 3, each mirror 11, 12 is  
2 provided with a separate housing 40, 41. The  
3 housings 40, 41 may further be contained in an outer  
4 housing, not shown, which may be partially or  
5 completely evacuated. The apparatus allows alignment  
6 of the second mirror 12 relative to the first mirror  
7 11 and translation of the second mirror 12 along the  
8 beam axis 43 by means of control mechanism 44.

9  
10 Alignment of the whole mirror assembly relative to  
11 the X-ray source 9 is possible by means of control  
12 mechanism 45. Mechanisms 44 and 45 are similar to  
13 mechanism 35 described with reference to Fig. 2, and  
14 are not described further.

15  
16 Although the invention has been described with  
17 reference to a microfocus X-ray generator, the  
18 invention can be used with any suitable X-ray  
19 generator which is capable of producing a small  
20 source of sufficient intensity.

21  
22 The mirror housing 30, 40 may be attached to the X-  
23 ray tube 1 or may be positioned independently.

24  
25 These and other modifications and improvements can be  
26 incorporated without departing from the scope of the  
27 invention.

## 1 CLAIMS

2

3 1. An apparatus for carrying out X-ray fluorescence  
4 spectrometry comprising an X-ray generating tube (1) and  
5 two paraboloidal X-ray reflecting mirrors (11, 12), the  
6 generating tube having an X-ray source (9) and an X-ray  
7 exit window (3) through which X-ray radiation from said  
8 source is emitted,  
9 the first mirror (11) being aligned on a first axis (32,  
10 43) and positioned in close coupled arrangement adjacent  
11 to the exit window (3), the second mirror (12) being  
12 aligned on said first axis and being positioned in spaced  
13 apart relationship to the first mirror (11),  
14 the first mirror (11) being adapted to collect diverging  
15 X-ray radiation at its first end adjacent to the  
16 collecting window (3) and to emit X-ray radiation in a  
17 substantially parallel beam at its second end,  
18 the second mirror (12) being adapted to collect  
19 substantially parallel X-ray radiation at its first end  
20 closest to the first mirror and to emit X-ray radiation  
21 in a focused beam at its second end.

22

23 2. An apparatus according to Claim 1, wherein the first  
24 and second mirrors (11, 12) are cylindrical specularly  
25 reflecting mirrors.

26

27 3. An apparatus according to Claim 1 or 2, wherein the  
28 first end of the first mirror (11) is positioned between  
29 5 and 50 mm from the X-ray source (9).

30

31 4. An apparatus according to any preceding Claim,  
32 wherein the apparatus further comprises a housing  
33 containing the first and second mirrors.

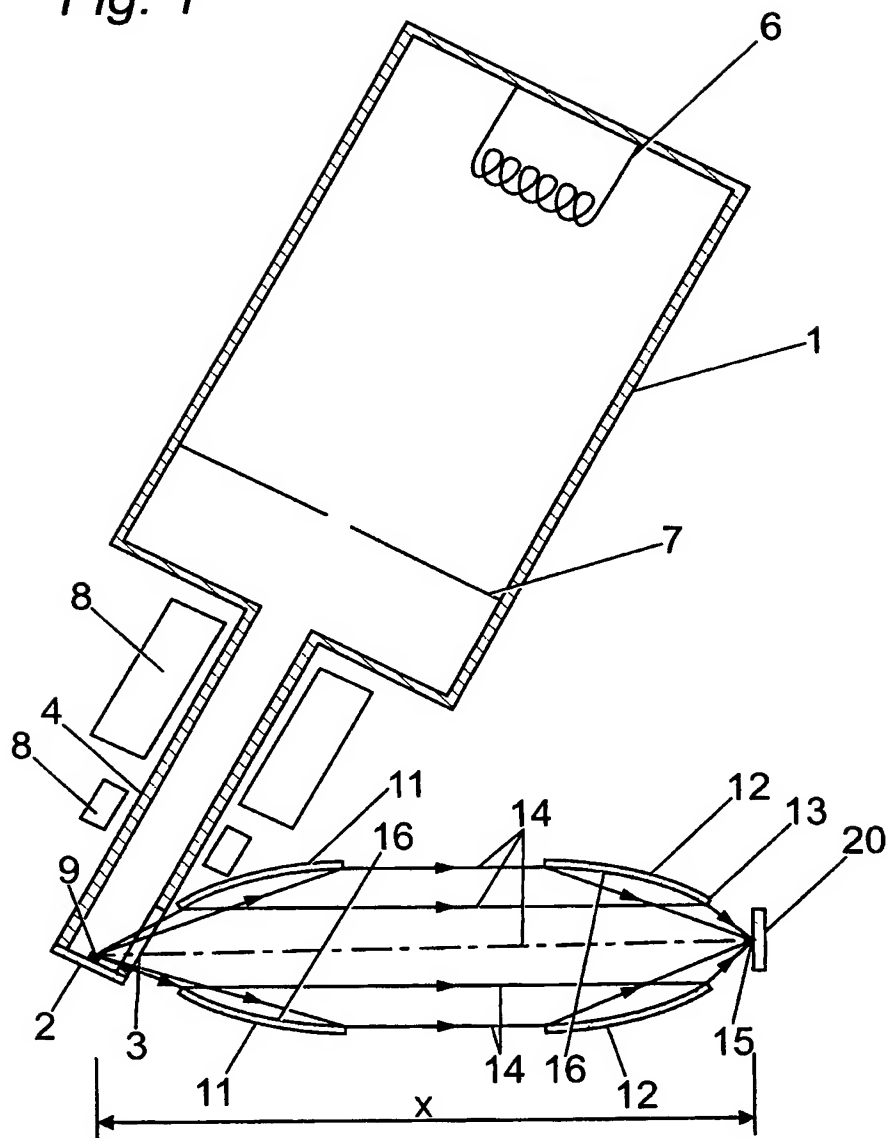
- 1  
2 5. An apparatus according to any preceding Claim,  
3 wherein the second mirror is fixed in position relative  
4 to the first mirror.  
5
- 6 6. An apparatus according to any of Claims 1 to 4,  
7 wherein the second mirror is movable in position relative  
8 to the first mirror.  
9
- 10 7. An apparatus according to Claim 6, further  
11 comprising a guide means for guiding said second mirror  
12 in a direction parallel to the first axis, and adjustment  
13 means for adjusting the spacing of the first and second  
14 mirrors.  
15
- 16 8. An apparatus according to any preceding Claim,  
17 further comprising angular adjustment means adapted to  
18 allow angular adjustment of the mirror housing with the  
19 X-ray generator tube.  
20
- 21 9. An apparatus according to any preceding Claim,  
22 wherein the X-ray generator tube is adapted to produce an  
23 X-ray source at the target having a maximum width of less  
24 than 50  $\mu\text{m}$ , more preferably less than 15  $\mu\text{m}$ .  
25
- 26 10. An apparatus according to any preceding Claim,  
27 wherein the apparatus is portable and the X-ray generator  
28 is a microfocus generator.  
29
- 30 11. A method of delivering X-ray radiation to a specimen  
31 for the purpose of X-ray fluorescence spectrometry using  
32 an X-ray generating tube, the generating tube having an

- 1 X-ray exit window through which X-ray radiation is  
2 emitted,  
3 the method comprising placing first and second  
4 paraboloidal X-ray reflecting mirrors between the exit  
5 window and the specimen,  
6 using the first mirror to collect diverging X-ray  
7 radiation at its first end adjacent to the exit window  
8 and to emit X-ray radiation in a substantially parallel  
9 beam at its second end,  
10 and using the second mirror to collect substantially  
11 parallel X-ray radiation at its first end closest to the  
12 first mirror and to emit X-ray radiation at its second  
13 end to a focused spot on the specimen.  
14
- 15 12. A method according to Claim 11, wherein the first  
16 and second mirrors (11, 12) are cylindrical specularly  
17 reflecting mirrors.  
18
- 19 13. A method according to Claim 11 or 12, wherein the  
20 first end of the first mirror (11) is positioned between  
21 5 and 50 mm from the X-ray source (9).  
22
- 23 14. A method according to any one of Claims 11 to 13,  
24 wherein the focused spot on the specimen has a maximum  
25 dimension of 50  $\mu\text{m}$ .  
26
- 27 15. A method according to any one of Claims 11 to 14,  
28 further comprising the step of adjusting the spacing of  
29 the first and second mirrors to produce a focused spot on  
30 the specimen.  
31
- 32 16. A method according to any one of Claims 11 to 15,  
33 wherein the X-ray generator is a microfocus generator.

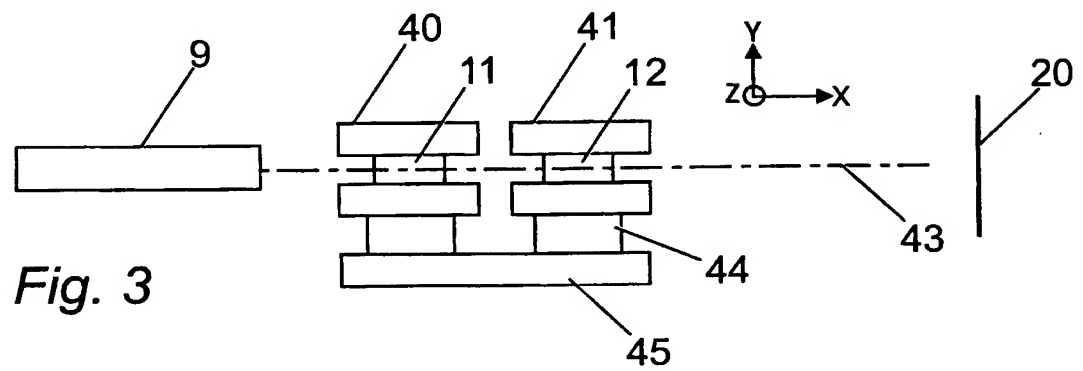
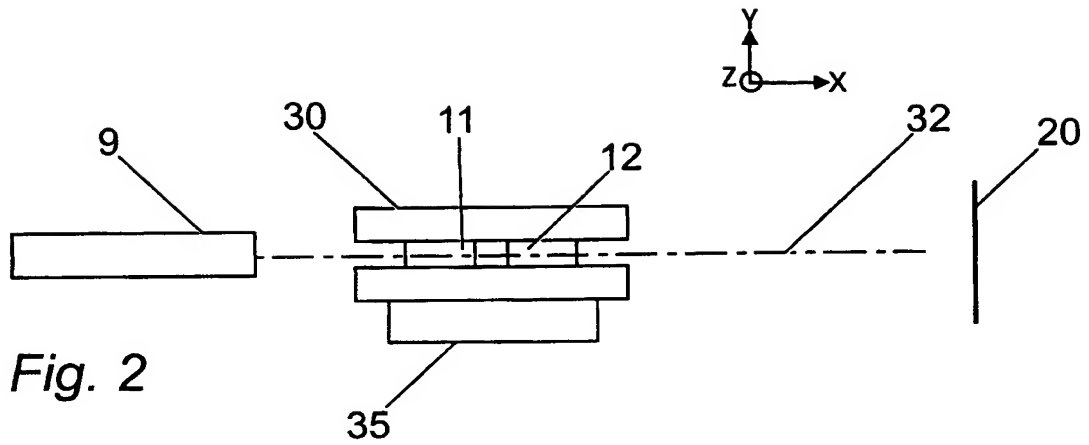


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Fig. 1



2 / 2



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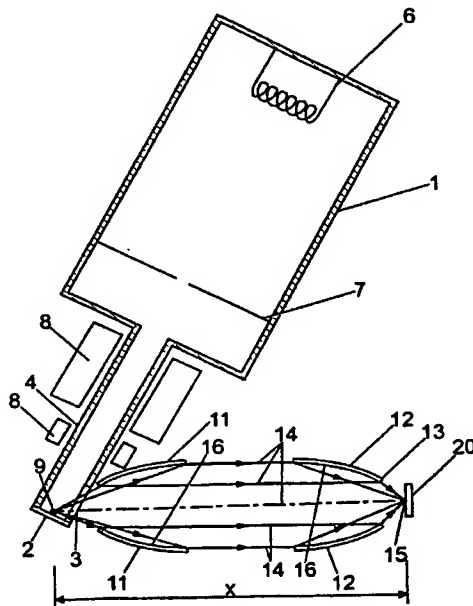
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(81) Designated States (*national*): AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

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(57) Abstract: This invention relates to a portable apparatus for carrying out X-ray fluorescence spectrometry on specimen materials at a distance from the apparatus. The apparatus comprises an X-ray generating tube (1), such as a microfocus tube, and two paraboloidal X-ray reflecting mirrors. The first collecting mirror (11) is positioned in close coupled arrangement adjacent to the exit window (3) of the tube (1), such that it emits parallel X-ray radiation (14) to the second focusing mirror (12) which is aligned on the axis of and spaced apart from the first mirror (11). The second mirror (12) collects the parallel X-ray radiation (14) at its end closest to the first mirror (11) and emits X-ray radiation in a focused beam onto the specimen (20). The distance between the first and second mirrors is adjusted to suit the distance from the X-ray tube (1) to the specimen (20). Focal spots on the specimen (20) of diameter less than 15  $\mu\text{m}$  are possible, enabling precise analysis of small areas of the specimen.

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patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

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7     distance from the apparatus.

8

9     X-ray fluorescence spectrometry is a non-destructive  
10    technique for determining the elemental composition  
11    of a wide variety of materials. X-ray fluorescence  
12    (XRF) is the secondary emission of X-rays at  
13    wavelengths characteristic of each element present  
14    when a material is irradiated with a primary X-ray  
15    beam. In commercially available XRF spectrometers  
16    the bulk sample is usually irradiated directly by X-  
17    rays from a sealed tube. The technique is  
18    sufficiently sensitive to detect elements which are  
19    present at concentrations as low as one or two parts  
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5 sometimes called Micro X-ray Fluorescence Analysis  
6 (MXRFA or MXA) apparatus.

7  
8 Several methods presently exist for MXRFA. Among  
9 them is the use of mono-capillary and poly-capillary  
10 X-ray focusing optics coupled to standard or  
11 microfocus X-ray generating tubes. These suffer from  
12 the drawback that samples have to be placed very  
13 close to the output of the optic (generally less than  
14 300  $\mu\text{m}$ ). The minimum focal spot generally  
15 commercially available with polycapillaries is 28  $\mu\text{m}$ .  
16 This is relatively large and limits the fineness of  
17 the resolution with which areas of a sample can be  
18 analysed.

19  
20 Another method which presently exists for MXRFA is to  
21 use a synchrotron in conjunction with Fresnel lenses.  
22 Such apparatus is massive and not portable, although  
23 beams having a focal spot of only 1  $\mu\text{m}$  can be  
24 achieved, giving greater accuracy in analysis of  
25 samples. This method suffers from the disadvantage  
26 that synchrotron radiation sources are large fixed  
27 facilities which are not portable and are not  
28 available in most laboratories, so cannot be accessed  
29 on a routine basis.

30

1 A further method of MXRFA which exists is the use of  
2 a synchrotron in conjunction with mono-capillary  
3 lenses. Such apparatus is also not portable, and  
4 beam sizes are limited to a focal spot of 5-10  $\mu\text{m}$ .

5

6 It is therefore an object of the invention to provide  
7 an apparatus for carrying out X-ray fluorescence  
8 spectrometry which is portable yet which is capable  
9 of analysing samples of are less than 30  $\mu\text{m}$ .

10

11 According to a first aspect of the present invention  
12 there is provided an apparatus for carrying out X-ray  
13 fluorescence spectrometry comprising an X-ray  
14 generating tube and two paraboloidal X-ray reflecting  
15 mirrors, the generating tube having an X-ray source  
16 and an X-ray exit window through which X-ray  
17 radiation from said source is emitted,  
18 the first mirror being aligned on a first axis and  
19 positioned in close coupled arrangement adjacent to  
20 the exit window, the second mirror being aligned on  
21 said first axis and being positioned in spaced apart  
22 relationship to the first mirror,  
23 the first mirror being adapted to collect diverging  
24 X-ray radiation at its first end adjacent to the  
25 collecting window and to emit X-ray radiation in a  
26 substantially parallel beam at its second end,  
27 the second mirror being adapted to collect  
28 substantially parallel X-ray radiation at its first  
29 end closest to the first mirror and to emit X-ray  
30 radiation in a focused beam at its second end.

31

1 By using first and second mirrors in this way, the  
2 focal spot on the target of the X-ray tube is  
3 transferred to the image plane, at unity  
4 magnification. The focal spot at the image plane on  
5 the sample subjected to fluorescence has a high  
6 brightness, and focal spots on the sample of diameter  
7 less than 15  $\mu\text{m}$  are possible.

8  
9 Preferably the first and second mirrors are  
10 cylindrical specularly reflecting mirrors.  
11 Preferably the first end of the first mirror is  
12 positioned between 5 and 50 mm from the X-ray source.

13  
14 Preferably the apparatus further comprises a housing  
15 containing the first and second mirrors.

16  
17 The second mirror may be fixed in position relative  
18 to the first mirror.

19  
20 Alternatively the second mirror may be movable in  
21 position relative to the first mirror. The apparatus  
22 may further comprise a guide means for guiding said  
23 second mirror in a direction parallel to the first  
24 axis, and adjustment means for adjusting the spacing  
25 of the first and second mirrors.

26  
27 The apparatus may further comprise angular adjustment  
28 means adapted to allow angular adjustment of the  
29 mirror housing with the X-ray generator tube.

30



1     Preferably the X-ray generator tube is adapted to  
2     produce an X-ray source at the target having a  
3     maximum width of less than 50  $\mu\text{m}$ , more preferably  
4     less than 15  $\mu\text{m}$ .

5  
6     According to a second aspect of the present invention  
7     there is provided a method of delivering X-ray  
8     radiation to a specimen for the purpose of X-ray  
9     fluorescence spectrometry using an X-ray generating  
10    tube, the generating tube having an X-ray exit window  
11    through which X-ray radiation is emitted,  
12    the method comprising placing first and second  
13    paraboloidal X-ray reflecting mirrors between the  
14    exit window and the specimen,  
15    using the first mirror to collect diverging X-ray  
16    radiation at its first end adjacent to the exit  
17    window and to emit X-ray radiation in a substantially  
18    parallel beam at its second end,  
19    and using the second mirror to collect substantially  
20    parallel X-ray radiation at its first end closest to  
21    the first mirror and to emit X-ray radiation at its  
22    second end to a focused spot on the specimen.

23  
24    Preferably the method uses an apparatus according to  
25    the first aspect of the invention.

26  
27    Embodiments of the invention will now be described,  
28    by way of example only, with reference to the  
29    accompanying figures, where:

30

1 Fig. 1 is a schematic view of two X-ray focusing  
2 mirrors used in accordance with the invention to  
3 focus an X-ray beam from the source on the X-ray  
4 target to the sample to be subject to X-ray  
5 fluorescence spectrometry;

6

7 Fig. 2 is a schematic view of an apparatus according  
8 to a first aspect of the invention having mirrors  
9 fixed relative to each other; and

10

11 Fig. 3 is a schematic view of an apparatus according  
12 to a second aspect of the invention having mirrors  
13 adjustable relative to each other.

14

15 Referring to Fig. 1 there is shown, in a schematic  
16 form and not to scale, an X-ray generating tube 1  
17 having an exit window 3, an electron source 6, an  
18 anode 7, focusing and stigmator coils 8 and a target  
19 2 on which is formed an X-ray source 9. A suitable  
20 X-ray generating tube is the MICROSOURCE™ tube  
21 described in International Patent Application No  
22 PCT/GB97/02022, which is a compact X-ray generator  
23 capable of producing small-size, high intensity X-ray  
24 sources for low power input. Typically the exit  
25 window 3 of the generator 1 is provided in the narrow  
26 portion 4 of the X-ray tube about which the X-ray  
27 focusing coils 8 are arranged, to the side of the X-  
28 ray target 2. A first X-ray focusing mirror 11, the  
29 collection mirror, is positioned adjacent to the exit  
30 window 3 in close coupled arrangement, and a second  
31 X-ray focusing mirror 12, the focusing mirror, is

1 arranged coaxially with the first X-ray focusing  
2 mirror 11, to transfer the . Suitable mirrors 11, 12  
3 are MICROMIRROR™ X-ray optics as supplied by Bede  
4 Scientific Instruments Ltd. The mirrors are  
5 cylindrical specularly reflecting mirrors. Each  
6 mirror comprises a cylindrical body having an axially  
7 symmetrical passage extending therethrough. There is  
8 an aperture at each end of the body which  
9 communicates with the passage. The reflecting  
10 surface is on the inside of the long axis of the  
11 cylinder and has a shape corresponding to a  
12 paraboloid of revolution about the long axis of the  
13 cylinder.

14

15 A paraboloidal profile produces an almost parallel,  
16 essentially non-divergent beam 14. The interior  
17 reflecting surface 16 is coated in an exceptionally  
18 smooth coating of gold or similar in order to provide  
19 specular reflectivity. Typically the mirror is made  
20 of nickel and is of the order of 10 to 100 mm in  
21 length, typically about 30 mm. The outside diameter  
22 of the mirror is typically 6 mm. The internal  
23 diameter is typically less than 4 mm. The entry  
24 aperture is generally smaller than the exit aperture.

25

26 The two mirrors have an identical profile. The  
27 source to first mirror distance is in the range 5 to  
28 50 mm.

29

1 Typically the X-ray generator produces a sub-15  $\mu\text{m}$   
2 spot source on a target of less than 10 mm diameter  
3 at a power of up to 30 W.

4  
5 The first mirror or paraboloidal optic 11 has a high  
6 angle of collection and reflects X-rays into a  
7 substantially parallel beam. In practice a beam of  
8 divergence less than 40 arc seconds can be achieved.

9  
10 The second mirror or paraboloidal optic 12 takes the  
11 parallel beam and focuses it down to a spot 15 on the  
12 specimen 20 of a size similar to that of the X-ray  
13 source, typically a spot with a diameter of less than  
14 15  $\mu\text{m}$ .

15  
16 The focus 15 of the second optic 12 is typically  
17 about 10 to 20 mm away from the far end 13 of optic,  
18 giving a much more convenient working distance than  
19 is available from prior art XRF apparatus, such as  
20 monocabillaries.

21  
22 The distance between the two optics 11, 12 may be  
23 continuously changed without affecting the focal spot  
24 quality, thereby allowing a range of source to sample  
25 distances X to be achieved. Typically distance X  
26 will be 100 mm or more.

27  
28 X-ray optics have very well defined profiles and low  
29 surface roughness, and therefore work at very high  
30 efficiency. By using paraboloidal mirrors the  
31 apparatus of the invention achieves broad band

1 transmission of X-rays, with an efficiency close to  
2 1, since only double reflection of the X-ray  
3 radiation is required.

4  
5 The invention achieves high X-ray brightness at the  
6 focal plane on the target, with a focal spot diameter  
7 of as low as 10  $\mu\text{m}$ .

8  
9 The apparatus of the invention is truly portable,  
10 giving it applications in areas such as forgery  
11 detection, which require the apparatus to be taken to  
12 the specimen.

13  
14 A parabolic surface will produce a parallel beam if  
15 the source is placed at the focal point. Conversely  
16 a focused beam will be brought to a focus when a  
17 parabolic surface is illuminated with a parallel  
18 beam. Therefore the method and apparatus of the  
19 invention serves to transfer the image of the X-ray  
20 spot from the target to the specimen. It should be  
21 noted that the target may not be perpendicular to the  
22 axis of the of the mirrors, so that the effective  
23 dimension of the image on the target, when viewed  
24 along the axis of the mirrors, is less than the  
25 actual dimension on the target.

26  
27 The focal spot size at the specimen is thus primarily  
28 determined by the spot size on the target of the X-  
29 ray tube. Since the first mirror produces a parallel  
30 beam, the focal spot size at the specimen is, within  
31 practical limits, independent of the distance of the

1 second mirror along the beam axis. Therefore the  
2 second mirror can be placed at the required distance  
3 from the first in order to suit the geometrical  
4 requirements of the equipment.

5  
6 Figs. 2 and 3 show two schematic arrangements for  
7 housing the apparatus of the invention.

8  
9 In the simplest case, shown in Fig. 2, the collector  
10 and focusing mirrors 11, 12 are aligned with each  
11 other and are fixed within a cylindrical housing 30.  
12 The housing is aligned relative to the X-ray source  
13 9, shown purely schematically in Figs. 2 and 3, on  
14 the beam axis 32, either fixedly or adjustably. The  
15 housing 30 may be subject to a partial or total  
16 vacuum, to improve the efficiency of the mirrors and  
17 reduce energy absorption as the X-rays pass through  
18 the gas in the housing 30. It is to be understood  
19 that in practice the source 9 is part of an X-ray  
20 generating tube 1 (not shown in Figs. 2 and 3).

21  
22 In use the housing 30 is placed adjacent to the X-ray  
23 source, and a control mechanism 35 allows fine  
24 adjustment of the position of the housing 30 in the  
25 x, y and z directions so that the axis 32 of the  
26 mirrors is accurately aligned with the X-ray source 9  
27 and directed to the specimen 20. The control  
28 mechanism 35 may comprise any suitable mechanisms  
29 which permit fine translational adjustment, such as  
30 lead screws or Vernier controls.

31

1 In the example of Fig. 3, each mirror 11, 12 is  
2 provided with a separate housing 40, 41. The  
3 housings 40, 41 may further be contained in an outer  
4 housing, not shown, which may be partially or  
5 completely evacuated. The apparatus allows alignment  
6 of the second mirror 12 relative to the first mirror  
7 11 and translation of the second mirror 12 along the  
8 beam axis 43 by means of control mechanism 44.

9  
10 Alignment of the whole mirror assembly relative to  
11 the X-ray source 9 is possible by means of control  
12 mechanism 45. Mechanisms 44 and 45 are similar to  
13 mechanism 35 described with reference to Fig. 2, and  
14 are not described further.

15  
16 Although the invention has been described with  
17 reference to a microfocus X-ray generator, the  
18 invention can be used with any suitable X-ray  
19 generator which is capable of producing a small  
20 source of sufficient intensity.

21  
22 The mirror housing 30, 40 may be attached to the X-  
23 ray tube 1 or may be positioned independently.

24  
25 These and other modifications and improvements can be  
26 incorporated without departing from the scope of the  
27 invention.

## 1 CLAIMS

2

3 1. An apparatus for carrying out X-ray fluorescence  
4 spectrometry comprising an X-ray generating tube (1) and  
5 two paraboloidal X-ray reflecting mirrors (11, 12), the  
6 generating tube having an X-ray source (9) and an X-ray  
7 exit window (3) through which X-ray radiation from said  
8 source is emitted,  
9 the first mirror (11) being aligned on a first axis (32,  
10 43) and positioned in close coupled arrangement adjacent  
11 to the exit window (3), the second mirror (12) being  
12 aligned on said first axis and being positioned in spaced  
13 apart relationship to the first mirror (11),  
14 the first mirror (11) being adapted to collect diverging  
15 X-ray radiation at its first end adjacent to the  
16 collecting window (3) and to emit X-ray radiation in a  
17 substantially parallel beam at its second end,  
18 the second mirror (12) being adapted to collect  
19 substantially parallel X-ray radiation at its first end  
20 closest to the first mirror and to emit X-ray radiation  
21 in a focused beam at its second end.

22

23 2. An apparatus according to Claim 1, wherein the first  
24 and second mirrors (11, 12) are cylindrical specularly  
25 reflecting mirrors.

26

27 3. An apparatus according to Claim 1 or 2, wherein the  
28 first end of the first mirror (11) is positioned between  
29 5 and 50 mm from the X-ray source (9).

30

31 4. An apparatus according to any preceding Claim,  
32 wherein the apparatus further comprises a housing  
33 containing the first and second mirrors.



1

2 5. An apparatus according to any preceding Claim,  
3 wherein the second mirror is fixed in position relative  
4 to the first mirror.

5

6 6. An apparatus according to any of Claims 1 to 4,  
7 wherein the second mirror is movable in position relative  
8 to the first mirror.

9

10 7. An apparatus according to Claim 6, further  
11 comprising a guide means for guiding said second mirror  
12 in a direction parallel to the first axis, and adjustment  
13 means for adjusting the spacing of the first and second  
14 mirrors.

15

16 8. An apparatus according to any preceding Claim,  
17 further comprising angular adjustment means adapted to  
18 allow angular adjustment of the mirror housing with the  
19 X-ray generator tube.

20

21 9. An apparatus according to any preceding Claim,  
22 wherein the X-ray generator tube is adapted to produce an  
23 X-ray source at the target having a maximum width of less  
24 than 50  $\mu\text{m}$ , more preferably less than 15  $\mu\text{m}$ .

25

26 10. An apparatus according to any preceding Claim,  
27 wherein the apparatus is portable and the X-ray generator  
28 is a microfocus generator.

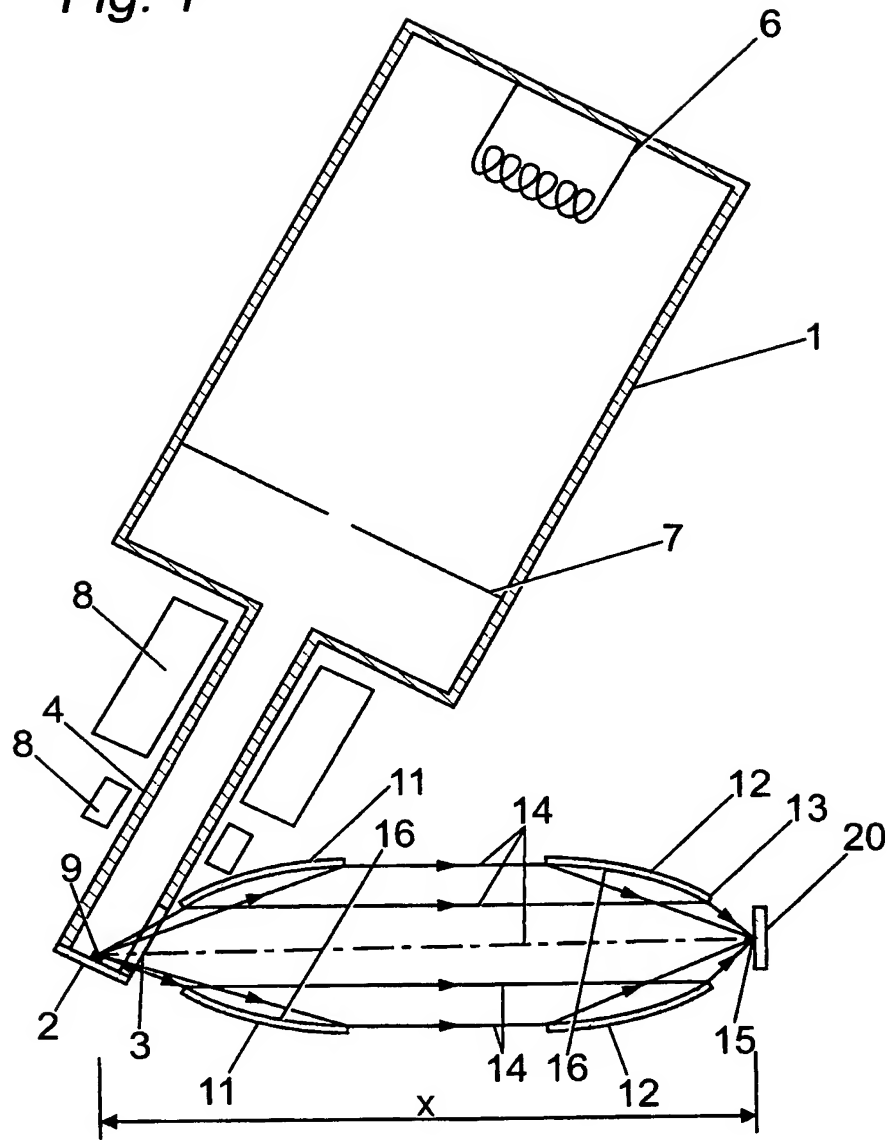
29

30 11. A method of delivering X-ray radiation to a specimen  
31 for the purpose of X-ray fluorescence spectrometry using  
32 an X-ray generating tube, the generating tube having an

- 1 X-ray exit window through which X-ray radiation is  
2 emitted,  
3 the method comprising placing first and second  
4 paraboloidal X-ray reflecting mirrors between the exit  
5 window and the specimen,  
6 using the first mirror to collect diverging X-ray  
7 radiation at its first end adjacent to the exit window  
8 and to emit X-ray radiation in a substantially parallel  
9 beam at its second end,  
10 and using the second mirror to collect substantially  
11 parallel X-ray radiation at its first end closest to the  
12 first mirror and to emit X-ray radiation at its second  
13 end to a focused spot on the specimen.  
14
- 15 12. A method according to Claim 11, wherein the first  
16 and second mirrors (11, 12) are cylindrical specularly  
17 reflecting mirrors.  
18
- 19 13. A method according to Claim 11 or 12, wherein the  
20 first end of the first mirror (11) is positioned between  
21 5 and 50 mm from the X-ray source (9).  
22
- 23 14. A method according to any one of Claims 11 to 13,  
24 wherein the focused spot on the specimen has a maximum  
25 dimension of 50  $\mu\text{m}$ .  
26
- 27 15. A method according to any one of Claims 11 to 14,  
28 further comprising the step of adjusting the spacing of  
29 the first and second mirrors to produce a focused spot on  
30 the specimen.  
31
- 32 16. A method according to any one of Claims 11 to 15,  
33 wherein the X-ray generator is a microfocus generator.

1 / 2

Fig. 1



2 / 2

